

REC'D 0 9 SEP 2003

WIPO

PCT

Patent Office Canberra

I, SMILJA DRAGOSAVLJEVIC, TEAM LEADER EXAMINATION SUPPORT AND SALES hereby certify that annexed is a true copy of the Provisional specification in connection with Application No. 2002951011 for a patent by NAUVEAU TECHNOLOGY INVESTMENTS LTD as filed on 27 August 2002.



WITNESS my hand this Third day of September 2003

5. Drougosavyene

SMILJA DRAGOSAVLJEVIC TEAM LEADER EXAMINATION SUPPORT AND SALES

PRIORITY DOCUMENT

SUBMITTED OR TRANSMITTED IN COMPLIANCE WITH RULE 17.1(a) OR (b)

S&F Ref: 605988

ORIGINAL

AUSTRALIA

Patents Act 1990

PROVISIONAL SPECIFICATION FOR THE INVENTION ENTITLED:

Water Treatment Process

Name and Address of Applicant:

Nauveau Technology Investments Ltd, c/o Moores Rowland Vanuatu, Windsor House, Port Vila, Vanuatu

Name of Inventor:

Daniel Maxwell Drew, Alfred Norman Ducksbury, Darryl Josey

This invention is best described in the following statement:

Water Treatment Process

Technical Field of the Invention

The invention relates to processes for reducing the concentration of soluble phosphorus species in water, especially wastewater.

Background of the Invention

Dissolved phosphorus is damaging to the aqueous environment because it, along with nitrogen, is a driver of organic growth. When aquatic growths capture inflowing nitrogen, phosphorus and other nutrients, the new growth settles, dies and releases its nutrients into the upper waters. These, and further inflowing nutrients, encourage repetition of the growth-regrowth cycle resulting in the silting up of the receiving body and subsequent ecological damage. This process of eutrophication by phosphorus is particularly disadvantageous to shallow fresh water bodies where growth is nutrient limited and the most influential nutrient is phosphorus.

Accordingly, authorities propose stringent limits on the discharge of total effluent phosphorus to surface waters. Typical limits for total effluent phosphorus vary, according to receiving body and local authority, in the range 0.1 - 1 mg/L. Total effluent phosphorus is the sum of the concentration of soluble phosphorus and the amount of phosphorus present in effluent suspended solids (expressed in units of mass/volume). The latter is the product of the amount of effluent suspended solids in the effluent, in units of mass/volume, and the fraction of phosphorus in the effluent suspended solids dry mass. Thus, for example, under typical discharge conditions, there may be 20 mg/L effluent total suspended solids containing 2.5 % phosphorus on a dry weight basis. In that case, the amount of phosphorus present in the effluent suspended solids is 0.5 mg/L.

It is therefore of critical importance that both suspended solids and soluble phosphorus are reduced to attain regulatory compliance and to avoid environmental damage.

Currently, soluble phosphorus is typically removed by precipitation of insoluble metal phosphates produced by reacting the soluble phosphorus with one or more metal ions, typically aluminium, iron and/or calcium. This prior art process for reducing the concentration of soluble phosphorus is described in *Biological and Chemical Systems for Nutrient Removal*; Water Environment Federation, Virginia, USA; Municipal Subcommittee of the Technical Practice Committee; 1998. Iron ions are the most commonly used because the physical properties of the precipitate are more amenable to

5

10

15

20

25

separation of the precipitate than when aluminium or calcium ions are used. Specifically, the use of iron produces less volume of sludge than when using aluminium, and the precipitate produced when calcium is used tends to be slimy and is produced only slowly.

With reference to the use of ferric ion, the reactions involved in the process are:

$$Fe^{3+} + H_2PO_4^- \rightarrow FePO_4 + 2H^+ \text{ and}$$

 $Fe^{3+} + 3H_2O \rightarrow Fe(OH)_3 + 3H^+$

Similar reactions apply to other metal ions that react to precipitate phosphorus.

The formation of metal hydroxides thus adds a competing reaction to the process of precipitation of metal phosphates and requires the addition of metal ions in excess of the stoichiometric amount indicated by the first reaction. The competing reactions also imply a lower limit to the residual soluble phosphorus remaining in the effluent. This lowest theoretically achievable concentration C_{Pres} is calculated as:

$$C_{Pres} = [H_3PO_4] + [H_2PO_4] + [HPO_4^{2-}] + [PO_4^{3-}] + [FeH_2PO_4^{2+}] + [FeHPO_4^{+}].$$

The theoretical value is a function of pH and can be demonstrated to be 0.04 mg/L at pH 6.8.

The overall reaction for the removal of phosphorus by Fe³⁺ can be written:

1.6 Fe³⁺ + H₂PO₄ + 3.8 OH
$$\rightarrow$$
 Fe_{1.6}(H₂PO₄)(OH)_{3.8}

and thus quantifies the stoichiometric Fe^{3+}/P molar ratio at which the theoretical minimum residual phosphate will be achieved as about 1.6 when the pH is such that the predominant soluble phosphorus species is H_2PO_4 , which is the case for most wastewaters.

However, the present best practice for soluble phosphate removal from treated effluent by ferric ion addition can only achieve a minimum residual soluble phosphorus of 0.06 mg/L at a molar ratio (Fe³⁺/P_{removed}) usually well above 4, and typically about 10. Present practice therefore requires at least 3 times and usually 6 times the theoretical stoichiometric amount of metal ions to compensate for pH variations which occur in practice to attain the minimum residual soluble phosphate concentration.

Accordingly, there is a need for a process for reducing the concentration of soluble phosphorus in water that utilises added metal ions more economically. A very desirable process would be one that enables the use of an amount of metal ions at or close to the stoichiometric amount and which is less sensitive to pH.

Existing water treatment processes exhibit a number of other shortcomings, which are described below.

Discharged effluent is typically subjected to regulation for the concentration of suspended solids, the limits imposed for total suspended solids being typically 20 mg/L.

5

10

15

20

25

30

In order to comply with these regulations costly separation procedures are undertaken. The complexity and cost of these procedures is greatly affected by the quantity and physical properties of the suspended solids. Typically such procedures as dissolved air flotation or belt filtration augmented by flocculation and polyelectrolytic coagulation are used. The cost of these coagulation and flocculation chemicals is substantial. Procedures that can modify the bulk density, filterability and sludge volume of the suspended solids can significantly reduce these costs and are thus economically advantageous. Furthermore, reduction in effluent suspended solids concentration will also markedly reduce the total effluent phosphorus concentration.

Additionally, certain metal ions are toxic to the ecosystem of the body receiving effluent water and are subject to regulation. In sewage treatment systems the operator typically controls the influent metal ion concentration by acceptance testing. However, as the limits for toxic metals are extremely low (the limits for most toxic metals are well below 10 mg/L) there is a need for economical removal processes, especially in the control of accidental contamination. Furthermore, the relevant authorities require the immobilisation of metal ions in sludges or solid wastes, which are to be discharged to the environment, as characterised by the Toxicity Characteristic Leaching Procedure (TCLP; USEPA Method 1311). Untreated sludges containing toxic metals may fail the TCLP test. Accordingly, there is a need for water treatment processes that produce sludges that pass the TCLP procedure.

Still further, in a typical wastewater treatment process the insoluble matter, which may be separated from the effluent liquid by any of a number of processes, is typically discharged to the environment, either as landfill, or for agricultural purposes, either alone or as a supplement in a composting process or other fertilising substance. After separation, the sludge typically develops disagreeable odours that are considered to be disadvantageous to the environment or for their proposed end use. Additionally, in a typical wastewater process, the effluent develops an odour during the treatment process and the odour may be discharged to the atmospheric environment in contravention of local regulation. The odour is typically produced by the biological production of organic and inorganic volatile sulfur compounds and is typically more evident as the effluent is stored on site or applied to land for irrigation purposes. There is therefore a need for a water treatment process that produces sludge and treated water that have reduced odour, or preferably no odour, and that do not develop odour over time.

It is an object of the present invention to at least partially satisfy one or more of the above mentioned needs.

10

20

30

Summary of the Invention

According to the invention, there is provided a process for reducing the concentration of dissolved phosphorus-containing species in water containing dissolved phosphorus-containing species, the process comprising the steps of

- (a) dispersing in said water an amount of a treating substance,
- (b) adding to said water an amount of at least one metal ion sufficient to at least partially precipitate a phosphorus-containing compound of said at least one metal, and
- (c) removing solids present in said water therefrom to produce a treated water; wherein said treating substance is selected from the group consisting of (i) bauxite refinery residue known as red mud, and (ii) red mud that has been at least partially reacted with calcium and/or magnesium ions so as to have a reaction pH, when mixed with 5 times its weight of water, of between 8.0 and 10.5.

Detailed Description of the Invention

In the process of the present invention, phosphorus is precipitated by the conventional metal ion chemical process in the presence of a treating substance that enhances the chemical efficiency of the process and improves the filterability of the resultant metal phosphate precipitate. The treating substance is either the bauxite refinery residue known as "red mud", or "red mud" that has been at least partially reacted with calcium and/or magnesium ions so as to have a reaction pH, when mixed with 5 times its weight of water, of between 8.0 and 10.5. Processes for the reaction of red mud with a solution of calcium and/or magnesium ions are described in International Patent Application No. PCT/AU01/01383, the contents of which are incorporated herein in their entirety, or they may involve the reaction of red mud with sufficient quantity of seawater to decrease the reaction pH of the red mud to the range of 8.0 to 10.5.

In summary, as taught in International Patent Application No. PCT/AU01/01383, a process for reacting red mud with calcium and/or magnesium ions may comprise mixing red mud with an aqueous treating solution containing a base amount and a treating amount of calcium ions and a base amount and a treating amount of magnesium ions, for a time sufficient to bring the reaction pH of the red mud, when one part by weight is mixed with 5 parts by weight of distilled or deionised water, to less than 10.5. The base amounts of calcium and magnesium ions are 8 millimoles and 12 millimoles, respectively, per litre of the total volume of the treating solution and the red mud; the treating amount of calcium ions is at least 25 millimoles per mole of total alkalinity of the red mud expressed as calcium carbonate equivalent alkalinity and the treating amount of

10

15

20

25

magnesium ions is at least 400 millimoles per mole of total alkalinity of the red mud expressed as calcium carbonate equivalent alkalinity. Suitable sources of calcium or magnesium ions include any soluble or partially soluble salts of calcium or magnesium, such as the chlorides, sulfates or nitrates of calcium and magnesium.

By the use of the treating substance in conjunction with ions of one or more metals capable of forming a precipitate of a phosphorus-containing compound, the present inventors have found that the amount of metal ions that needs to be added to the water to reduce the dissolved phosphorus concentration to about the theoretical limit is at, or close to, the stoichiometric amount, in contrast to currently known methods in which considerably more metal ions are required, as noted above. The quantity of treating substance required to obtain this benefit in the amount of metal ions added has been found, surprisingly, to be essentially independent of the initial concentration of dissolved phosphorus in the water. The amount of treating substance used is thus not critical to the present process. For example, the amount of treating substance can be about 1 g/L or more of water to be treated, but will more usually be not more than about 0.5 g/L, still more usually not more than about 0.3 g/L, even more usually up to about 0.25, 0.2, 0.15 or 0.1 g/L yet more usually up to about 50 mg/L. Typically, the amount of treating substance added will be about 50 mg/L, though beneficial effects on removal of phosphorus can be seen with additions of as little as 10 mg/L.

In step (b) of the process of the invention, the metal ion is typically at least one of iron, aluminium and calcium, still more typically iron, which may be ferric or ferrous iron or a mixture of the two. The amount added is typically not more than 1.5 times the stoichiometric amount required to react with the amount of dissolved phosphorus present, but an excess could be added if so desired. Suitably, the metal ion is added as a soluble salt of the metal such as a chloride, sulfate or the like.

Step (c) of the process of the invention may comprise any suitable procedure for the removal of solids from the treated water, and will typically be preceded by settlement of the precipitated phosphorus-containing compound(s) and any other solids present, suitably until the supernatant water is clear. One or more flocculating agents may be added, if so desired.

Optionally, the process of the invention may include the additional step of adjusting the pH of the water before step (b). Typically, the pH of the water is adjusted, if necessary, to a pH in the range of about 6.5 to 7.5.

5

lO

15

20

25

Because the treating substance is substantially insoluble in water and is easily dispersed throughout the body of the water it is believed that its presence modifies biosolids that may be present in the water in such a manner as to:

- improve filterability this improves the economics of the treatment process by minimising the need for filter aids, flocculants, and highly technical filtration apparatus, and reduces residual phosphate concentration by more complete removal of phosphorus-containing biosolids;
- improve the removal of precipitated insoluble inorganic phosphorus compounds by increasing the efficiency of the reaction between certain metal ions and phosphorus and by reducing the pH effect of that reaction;
- eliminate time dependent release of phosphorus from biosolids by stabilising the organic and inorganic solids;
- eliminate odour in effluent and separated biosolids;

5

01

15

20

25

30

eliminate post treatment odour generation in separated biosolids and effluent:

Without wishing to be bound by theory, the inventors speculate that these properties are imparted to the solids by interaction with the treating substance at the liquid-solid interface of the dispersed particles of the treating substance and are probably related to the mineralisation and particle size distribution of the treating substance.

The inventors further postulate the following mechanistic interpretation of the observed properties of the treating substance applied to the treatment of wastewater.

- 1. Treating substance is added to the wastewater, disperses and remains suspended for a finite period.
- 2. Odorous molecules such as H_2S , methyl mercaptan and other thiols and sulphides interact at the treating substance-wastewater interface and are effectively removed from solution.
- 3. Other inorganic ions (such as metal ions, phosphate ions and hydroxyl ions) migrate to the treating substance particles and remain in a more or less ordered form adjacent to the mineral structure of the treating substance.
- 4. Colloidally suspended biosolids in the solution are attracted to the treating substance particles (perhaps by virtue of particle charge attraction) and agglomerate.
- 5. Ferric ions (or other metal ions) are added and react with the phosphate and hydroxyl ions, at or in the vicinity of the treating substance particles which then act as nuclei around which ferric hydroxide and ferric phosphate flocs agglomerate.

6. The resultant increase in particle size of the suspended solids (agglomeration) causes the suspended matter, ferric hydroxide, ferric phosphate, treating substance, to settle rapidly resulting in improved filterability.

The process of the present invention is applicable to the treatment of any soluble phosphorus-containing water, including all soluble phosphorus-containing wastewater, especially where the effluent is discharged to a shallow slow moving freshwater receiving body. The process of the present invention is especially applicable to the treatment of municipal sewage. Examples of water that may be treated by the process of the present invention include raw sewage, effluent from a primary, secondary, biological nitrogen removal, or other sewage sedimentation or clarification plant and wastewater from any industrial or agricultural process which contains inorganic or organic soluble phosphorus.

The process of the present invention may be carried out at any stage in the wastewater treatment process, whether it be physicochemical or biological. It may be used on untreated, fresh sewage (influent) or at any stage within a wastewater treatment plant. However, it is more economically carried out after primary sedimentation and clarification is completed, and preferably after secondary treatment and clarification is completed. Specifically, in the case of sewage treatment, the process of the present invention is preferably, but not necessarily, carried out after secondary clarification and nitrogen reduction has been completed. The process of the present invention may be carried out in either aerobic or anaerobic conditions.

The process of the present invention reduces the concentration of all forms of soluble phosphorus.

The process of the present invention exhibits the additional advantage that, if the water to be treated additionally contains one or more metals in excess of approved discharge concentrations, the concentration of the metals remaining in the solution after it has been treated by the process of the present invention is typically substantially reduced, usually to a level below applicable discharge limits. This is particularly advantageous if the metal is toxic to the ecosystem of the receiving body or is toxic to humans. Metals that may be substantially removed from water in this way include arsenic, cadmium, chromium, copper, lead, mercury, nickel and zinc. In the process of the present invention, metals present in the water to be treated are removed in the solid phase that is separated from the treated water in step (c), and are substantially immobilised in the solid phase so that the solids typically comply with the Toxicity Characteristic Leaching Procedure.

5

10

15

20

25

The use of the treating substance in the process of the present invention, in addition to enabling water to be treated with significantly lower amounts of metal ions such as iron ions (compared to prior art processes), facilitates separation of the solids precipitated when the metal ions are added to the water and thus allows water to be treated with the use of substantially reduced amounts of filter aids, flocculating agents, and the like, which are typically called for in prior art processes. The presence of the treating substance in the separated solids improves their bulk density and particulate nature and reduces their moisture content, compared to prior art processes, and thus reduces the effluent residual suspended solids content. Typically, the suspended solids content of the treated water produced by the process of the present invention is substantially less than 20 mg/L.

Additionally, when the solids removed in step (c) of the process of the invention are combined with the underflow from primary or secondary sedimentation processes, they similarly enhance the physical properties of the combined inorganic and organic sludges so that the efficiency of the polyelectrolyte flocculants used in the conventional solids separation is significantly improved.

Furthermore, it has been found, surprisingly, that neither the treated water produced by the process of the present invention, nor the sludge (solids) separated from the treated water develops an odour over time, such as when the sludge is disposed of as landfill or when the sludge or the treated water are used for agricultural application such as a soil supplement, as a supplement to a composting process, or for irrigation. In particular, the treated effluent may be stored without further odour development for extended periods if so desired. The separated solids that contain the treating substance and precipitated inorganic phosphorus compound(s), furthermore, have the property that when added to biological wastes that have a propensity to develop odour on storage or use, they inhibit that propensity.

In one particular form of the invention, therefore, there is provided a process for eliminating and preventing the redevelopment of odour from biosolid sludges separated from aqueous wastewater by clarification, settling, and separation, in which the solids removed in step (c) of a process according to the invention are combined with the biosolids underflow from a wastewater treatment plant prior to dewatering. This process is particularly applicable to biosolids from a sewage treatment process.

In summary, as a result of the application of the process of the invention the following benefits may be achieved:

5

25

- the amount of metal ion chemical required for maximum removal of phosphorus is reduced from the conventionally experienced molar ratio of metal to phosphorus of about 10 to a value of less than 2, typically in the range of 1.4-2.
- the pH range for the lowest theoretically achievable residual phosphorus concentration is markedly extended,
- the amount of polyelectrolyte required for solids separation is reduced substantially, typically by about 50%,
- the effluent suspended solids is reduced below 20 mg/L,
- the total effluent phosphorus (organic and inorganic phosphorus) is reduced below
 0.5 mg/L,
 - the development of odour in the effluent is inhibited,
 - the development of odour in the sludge is inhibited,
 - the filterability of the sludge is improved,

5

10

20

25

30

- time-dependent release of phosphorus from biosolids is eliminated,
- the toxic metals concentration in the effluent is reduced, and
 - the toxic metals in the separated solids and suspended solids are immobilised.

The treating substance that contributes the particular advantageous properties to the process of the invention is preferably a material obtainable from Virotec International Pty Ltd, of Sanctuary Cove, Queensland, Australia, under the trademark Bauxsol.

Examples

The following Examples are included to illustrate the invention, but they are not intended to place any limitation on the scope of the invention.

Example 1: Laboratory scale experiments on water containing phosphorus ions

A simulated phosphorus containing wastewater was prepared, consisting of an aqueous solution of potassium dihydrogen phosphate containing 6.09 mg/L phosphorus.

Procedure

To 200 ml samples of the phosphorus containing water was added ferric chloride in amounts corresponding to 0.84, 0.94, 1.12, 1.40 and 1.87 times the stoichiometric requirement for the complete precipitation of phosphorus as the insoluble ferric phosphate compound and, after 10 min, the pH of the resultant solution was adjusted to pH 6.5 - 7.5 with sodium carbonate. The solutions were filtered through a 0.47 micron filter and the filtrate was analysed for pH and phosphorus (Ascorbic Acid Method 4500-P E, Standard

Methods For the Examination of Waters and Waste Waters, 19th Edition, 1995, APHA AWWA WEF. 4-113, 5). Analytical results below 0.05 mg P/litre were confirmed by ion

chromatography. The level of detection of this procedure is 0.01 mg P/litre and the reproducibility was determined by replicate determinations to be \pm 0.02 at 95% confidence.

The above experiments were concurrently repeated by adding 10, 20 and 50 mg/L BauxsolTM 10 minutes prior to the ferric chloride additions.

The measured phosphorus concentrations were compared with the theoretical minimum concentration for the pH at which the precipitation was measured to occur.

The results are presented in Tables 1.1 to 1.5.

Table 1.1: Phosphorus (mg/L) determined after 2 hours

Fe/P	Bauxsol (mg/L)				
	0	10	20	50	
0.84	1.36	1.15	1.07	0.73	
0.94	1.61	0.93	0.79	0.73	
1.12	0.73	0.51	0.41	0.25	
1.40	0.38	0.04 0.23		0.04	
1.87	0.04	0.08	0.03	0.14	

Table 1.2: pH after phosphorus determination

Fe/P 0.84	Bauxsol (mg/L)				
	0	10 .	20	50	
	7.00	6.82	6.78	7.43	
0.94	7.80	7.13	7.48	7.68	
1.12	7.60	7.68	7.72	7.65	
1.40	7.64	7.55	7.62	7.65	
1.87	7.60	7.52	7.56	7.67	

It can be seen from Table 1.1 that, in the presence of BauxsolTM, the ferric chloride reaction procedure consistently removes phosphorus from the wastewater to a level significantly below the minimum theoretical level for the conventional ferric chloride process, and to a level below that obtainable through the use of ferric chloride alone, except for relatively large additions of ferric chloride.

Further, although the ferric chloride reaction is reportedly adversely affected by pH outside the range of pH 6.8 - 7.2, the presence of BauxsolTM enables the reaction to proceed outside that range. This pH phenomenon therefore considerably reduces the risk of treatment failure resulting from unexpected changes in wastewater pH.

It is known that in the ferric chloride reaction with ions of phosphorus species, the minimum residual phosphorus concentration is related to the pH at which the precipitation occurs. Thus comparisons of efficiency must be made at constant pH.

Accordingly, Table 1.4 compares the measured residual phosphorus concentration against the theoretical value at the same pH (shown in Table 1.3). In this comparison values above 1 indicate that incomplete precipitation has occurred. However Table 1.4 clearly illustrates that, at BauxsolTM levels of 10 mg/L and above, complete precipitation of phosphorus is achieved at Fe/P molar ratio between 1.1 and 1.4 whereas in the absence of BauxsolTM complete precipitation requires Fe/P molar ratio substantially greater than 1.4.

Table 1.3: Theoretical minimum residual phosphorus at pH of precipitation

Fe/P	Bauxsol (mg/L)					
	0	10	20	50		
0.84	0.04	0.04	0.04	0.14		
0.94	0.33	0.05	0.18	0.28		
1.12	0.23	0.28	0.30	0.25		
1.40	0.25	0.20 0.24		0.25		
1.87	0.23	0.19	0.21	0.28		

Table 1.4: Ratio of measured phosphorus to theoretical minimum phosphorus

Fe/P	Bauxsol (mg/L)					
	0	10	20	50		
0.84	34.00	28.75	26.75	5.21		
0.94	4.88	18.60	4.39	2.61		
1.12	3.17	1.82	1.37	1.00		
1.40	1.52	0.20	0.96	0.16		
1.87	0.17	0.42	0.14	0.50		

The values calculated in Table 1.5 represent the phosphorus concentration to be expected if the reactions were all completed at pH 6.8-7.0. The values were obtained by interpolation of the analytical data into the graphical data of Fig 3.2 of *Biological and Chemical Systems for Nutrient Removal*; Water Environment Federation, Virginia, USA; Municipal Subcommittee of the Technical Practice Committee; 1998 and, for the values less than 0.04 mg/l, are limited in significance by the reproducibility of the method. However the method has demonstrated a lower detection limit of 0.01 mg/L and Tables 1.1, 1.4 & 1.5 support a tentative conclusion that the presence of BauxsolTM may allow

10

15

residual phosphorus concentration less than the theoretical minimum value possible with the ferric ion reaction.

Table 1.5: Phosphorus concentration corrected for pH after phosphorus determination

Fe/P	Bauxsol mg/L					
, -	0	10	20	50		
0.84	1.36					
0.94	0.20	0.08	0.19	0.11		
1.12	0.13	0.07	0.05	0.04		
1.40	0.06	0.01	0.04	0.01		
1.87	0.01	0.02	0.01	0.03		

Example 2: Laboratory scale treatment of raw sewage influent containing phosphorus

Procedure

10

15

20

To 1000 ml samples of phosphorus-containing raw sewage was added ferric chloride in amounts approximating the stoichiometric requirement for the complete precipitation of phosphorus as the insoluble ferric phosphate compound. The solutions were allowed to settle and the supernatant liquor was analysed for pH and phosphorus. The level of detection of this analytical procedure was 0.03 mg P/litre.

In these experiments the sludge volume of the precipitated ferric phosphate and biosolids was estimated. The above experiment was concurrently duplicated by adding 87 mg/l BauxsolTM, 10 minutes prior to the ferric chloride additions.

The results are presented in Table 2.

Table 2

Fe/p Stoichiometric	Bauxsol TM mg/L	Residual Phosphorus mg/L	рН	Sludge Volume %		
0	0 -	12.4	7.85	n.a.		
0.75	0	7.5	7.1	10		
1.5	0	1.95	6.3	10		
1.0	87	Not detected	6.9	4		

In this experiment BauxsolTM was added at a rate of 87 mg/L although it was subsequently discovered that increasing BauxsolTM above 50 mg/L has no effect on the process. These experiments indicate that in raw sewage the addition of BauxsolTM to the conventional ferric ion precipitation process for the removal of phosphorus ions removed phosphorus from the wastewater to a level below the detection level and significantly

below the minimum theoretical level for the conventional ferric chloride process. Further, the sludge volume of ferric hydroxide, ferric phosphate and biosolids in the presence of BauxsolTM is shown to be approximately 40 % of the volume produced by ferric chloride alone.

Example 3: Laboratory scale treatment of odorous biosolids from raw sewage

Procedure

10

20

25

30

Biosolids from Pine Rivers STP were treated with either BauxsolTM alone or BauxsolTM/iron phosphate sediment collected from treatment of final effluent.

The ratio of solids in the mix was 1:3 on a dry weight basis (i.e. 25% Bauxsol additive to biosolids.)

Odour species characterisation

10g of untreated biosolids was placed in a headspace vial and sealed. 10g of treated biosolids was sealed in another vial. The headspace air composition in the two vials was analysed using GCMS and GC-Flame Photometric detector (which is specific for S-compounds).

In the untreated biosolids vial the species identified were:

Hydrogen Sulphide >2000 ppm

Methyl Mercaptan 100 ppm

Thiols & sulphides Trace

Dimethyl sulphide 1 ppm

In the treated biosolids vial the species identified were:

Dimethyl sulphide 1ppm

Both vials were analysed periodically over a number of weeks and although the untreated biosolids vial continued to generate odorous species, the treated biosolids vial

contained only the low level dimethyl sulphide species which is substantially odourless.

It was subjectively observed that the untreated biosolids emitted a characteristic odour consistent with that of hydrogen sulphide, mercaptans and thiols, whereas the treated biosolids were almost odourless.

This experiment illustrates the effect of BauxsolTM in the removal of odorous substances from wastewater and wastewater biosolids and in the inhibition of the development of odours with aging of the treated biosolids and wastewater treated effluent.

Example 4: Pilot Plant treatment of wastewater containing phosphorus and suspended solids

Procedure

10

15

20

25

30

1000 litres of raw sewage containing 13 mg/L of phosphorus at pH 7.65 was treated in the following sequence.

- 1. Add 100 mg/L BauxsolTM.
- 2. After 10 min add 100 mg/L ferric chloride (FeCl₃.6H₂O), 30 mg/L ferrous sulfate (FeSO₄.7H₂O) and 5 mg/L ferric sulfate (Fe₂(SO₄)₃.9H₂O). (This is approximately 1.2 times the stoichiometric requirement for P precipitation.)
 - 3. Allow to settle for 2 hours.
 - 4. Harvest.

The process achieved a residual phosphorus concentration <0.07 mg/L and it was noted that the suspended solids agglomerated almost instantaneously and settled rapidly.

This process was carried out with Technical grade chemicals in a manner consistent with commercial operations and demonstrates that the procedure achieves close to minimum theoretical residual phosphorus at or near the minimum stoichiometric metal ion requirement and that the physical nature of the suspended solids was modified in a beneficial way.

Example 5: Pilot plant treatment of partially treated wastewater containing phosphorus and suspended solids

Procedure

1000 litres of treated water overflow from a secondary clarifier post BNR treatment containing 5:55 mg/L phosphorus was treated in the following sequence

- 1. Add 50 mg/L BauxsolTM.
- 2. After 10 min add 50 mg/L ferric chloride 90% (1.5 times the stoichiometric requirement for P precipitation).
 - 3. Allow to settle for 2 hours.
 - 4. Harvest.

The process achieved a residual phosphorus concentration <0.07 mg/L.

This process was carried out with Technical grade chemicals in a manner consistent with commercial operations and demonstrates that the procedure achieves close to minimum theoretical residual phosphorus at or near the minimum stoichiometric metal ion requirement.

Example 6: Laboratory scale measurements of sludge volume on water containing phosphorus ions

Procedure

10

To 200 mL phosphate solution, in measuring cylinders, was added ferric chloride in amounts approximating the stoichiometric requirement for the complete precipitation of phosphorus as the insoluble ferric phosphate compound. The sludge volume of the precipitated ferric phosphate and hydroxide was measured at 10, 30 and 60 minutes after addition of the precipitants. The above experiment was concurrently duplicated by adding 50 mg/L BauxsolTM, 10 minutes prior to the ferric chloride additions.

The results are presented in Table 3

Table 3

	Bauxsol and ferric chloride		Ferric chloride only			Sludge volume ratio			
Fe/P	Sludge Volume in mL.								
	10 min	30min	60min	10 min	30min	60min	10 min	30 min	60 min
0.84	19	16	12	17	11	10	1.12	1.45	1.20
0.94	14	10	10	9	S	8	1.56	1.25	1.25
1.12	14	10	10	33	18	15	0.42	0.56	0.67
1.40	16	12	11	19	14	14	0.84	0.86	0.79
1.59	20	15	14	13	12	12	1.54	1.25	1.17
1.87	16	13	13	14	13	13	1.14	1.00	1.00

It can be seen that in the region of interest (1.0 - 1.5 times stoichiometric iron addition) the presence of BauxsolTM reduces the initial sludge volume by 40 - 50% and also increases the initial incremental settling rate.

Dated 27 August, 2002
Nauveau Technology Investments Ltd
Patent Attorneys for the Applicant/Nominated Person
SPRUSON & FERGUSON